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ANALYSIS OF SPICES.

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ONE of the difficult classes of vegetable substances to analyze satisfactorily is the class known as the spices. The quality of food accessories and their value for medicinal use resides mainly in two constituents—an aromatic volatile oil, and a resin, oftentimes pungent in character. In the case of ginger we have a volatile oil, 1 to 3 per cent, which imparts the odor and flavor of the drug, and a non-volatile resino-extractive to which the pungency is due. To these two principles the ginger owes its special activity. The constituents of ginger that may be neglected practically in chemical analysis are starch, fat and other inert principles, but starch is an important constituent in microscopical analysis.

The volatile constituent above referred to is readily extracted and separated from inert material by alcohol, ether, benzol, or by carbon disulfid. From good ginger ether extracts from 4 to 8 per cent. of an oleoresin, which contains the volatile oil and pungent principles. The ash from good ginger amounts to from 4 to 5 per cent. The presence of a very minute quantity of an alkaloid has been noted, but in estimations of quality this possible constituent is ignored.

According to the provisional methods for the analysis of spices in general, the Association of Agricultural Chemists recommends that the following data be obtained: Percentage of moisture, percentage of ash, of water-soluble ash, of ash insoluble in acid, of lime, of nitrogen, determination of ether extract (volatile and non-volatile), of alcoholic extract, of reducing matters, of starch, of crude fiber, and of tannin. For extracting ginger the percentage yield of cold-water extract is obtained, and for mustard the percentage of total sulfur, beside the microscopical examination. It will be seen that this process is quite an elaborate one if all these data are obtained. In certain individual spices only a few of these are essential, as in the case of ginger the United States standard requires that it shall contain not less than 42 per cent. of starch, not more than 8 per cent. of crude fiber, not more than 6 per cent. of total ash, not more than 1 per cent. of lime, and not more than 3 per cent. of ash insoluble in hydrochloric acid. In the limed or bleached ginger the standard is: It should contain not more than 10 per cent. of ash, not more than 4 per cent. of carbonate of lime, and conform in every respect to the above standard. It is clear that in order to obtain these data considerable time and labor are required, and when one has to run through a set of several dozen samples it involves an expenditure of time and labor which is, in some cases, unnecessary, and is certainly uneconomical from the point of view of those who have to bear the expense of the time, the expert help, and the material consumed.

It becomes, therefore, desirable that a rapid method, as a preliminary one for quickly separating the substandard and standard material, be employed. A somewhat indirect method of such a character has been suggested by E. Beckmann, which he calls the cryoscopic method. Mr. Beckmann reviews the difficulties the other processes involve, and these difficulties led him to devise one as above characterized. He based the estimation of spices on the rapid determination of the percentage of volatile-oil constituent. The solvent employed for the extraction of the volatile oil is ethylene bromid, on account of its high molecular depression. The examination is performed as follows: Five grams of the powdered drug are macerated in an Erlenmeyer flask for one day with 30 gm. of anhydrous ethylene bromid. As the drug floats on the surface of this liquid it is completely exhausted without much shaking in from eight to ten hours. The solution is filtered through wool, with the aid of the filter-pump, directly into the freezing tube or apparatus, and the freezing-point determined in the usual way, the bath being maintained at 5° to 6° C. The depression of the freezing-point below that of ethylene bromid observed is due partly to the volatile oil and partly to other substances dissolved by the ethylene bromid, and to obtain the former alone a second experiment must be made with a portion (5 gm.) of the drug freed from volatile oil by driving through it a current of steam at 130° C. The drug after this treatment is quite dry and can be used at once for the second extraction. The difference between the depression observed for the crude spice and for the spice deprived of volatile oil gives the depression due to the volatile oil. For estimating the quality of a spice by comparison with one of known standard strength, it is only necessary to make a determination using the standard as one, and another using the sample example to be estimated.

While Beckmann's method is comparatively short and rapid, it is less elaborate than process of extraction with petroleum spirit or ether and estimation of volatile ether extract. It has some of the disadvantages of the volatile ether extract method, in that it bases the estimation mainly on the volatile oil. In many cases the pungent resin or oleoresin is a very important ingredient of the spice, and we feel that this pungent substance should be considered in the preliminary test.

In order to quickly eliminate substandard material among the spices it has seemed to us that if the examination utilized the value of sense of taste, applying it to certain standard solutions, it would go far toward accomplishing the result sought. It has been determined that the sense of taste is even more acute and delicate than chemical analysis seems to be. It has been stated by certain analytical chemists that they were so positive of their results in the determinations they made by using the sense of taste or odor that they were willing to testify in court having no other basis for the testimony than this. One of our famous chemists, especially expert in the examination of unsanitary waters, makes the statement that they can unerringly detect certain forms of algæ in the water by odor alone, previously heating the same to a certain degree. So positive are the results that they are considered sufficient for court cases. Another one of our most able chemists, who is especially expert in the analysis of vanilla preparations, makes the statement that he considers no one an expert analyst for vanilla preparations unless he is able to detect minute quantities of tonka beans, if any have been added to the vanilla extract. Tea specialists depend entirely on the sense of taste, and they become so expert it is well known that they can grade the article with accuracy.

Prof. E. H. S. Bailey has ascertained that the delicacy of taste is so remarkable that an average person would detect one part of quinine in 500,000 of a solution. He has ascertained, also, that this delicacy of taste is to be considered in the following order: (1) bitter; (2) sweet; (3) acid; (4) alkaline; (5) saline. The order of delicacy for aromatics was not determined.

One of the first to recognize the value of the sense of taste in estimating the quality of a drug was Dr. E. R. Squibb, who suggested a physiological test for the standarization of tincture of aconite, which was about as follows: In the absence of any reliable chemical tests for aconitine E. R. Squibb suggested that a fluid drachm of a highly diluted solution of the various preparations be taken into the anterior part of the mouth (after the latter had been thoroughly rinsed) and held there one minute by the watch, and then discharged. The peculiar numbing sensation charcteristic of aconite should be experienced within fifteen minutes, and it should continue for fifteen or thirty minutes. Tested in this way, he found he could estimate the values of the various commercial aconitines.

He found, for example, that to be a standard 1 grain of good powdered aconite root is equal to $\frac{1}{8}$ grain of Merck's ordinary aconitine, $\frac{1}{38}$ grain of Merck's pseudaconitine, $\frac{1}{111}$ grain of Duquesnel's crystallized aconitine (really aconitine nitrate). He also found by this approximate method that 1 grain of powdered aconite root was equivalent to 1 minim of fluid extract, $\frac{1}{6}$ grain of alcoholic extract of aconite root, 2.66 minims of U. S. (1890) or 9.31 minims of U. S. (8th revision) tincture of aconite root, 8.43 minims of British tincture of the root, 11.8 minims of German tincture of the root, 1.5 minims of Fleming's tincture, 9 grains of powdered aconite leaf, 1.5 grains of alcoholic extract of dried aconite leaf, 1 grain of Allen's English extract of fresh plant, and 72 minims tincture of aconite leaf.

To employ this physiological method, employing sense of taste in the estimation of spices, an alcoholic extraction of definite strength of the aromatic and pungent principles should be made and this solution diluted to a maximum with water, the latter added to the limit of precipitation. The solution is then brought in contact with the gustatory nerve endings, and in order that a comparison be made a uniform dilution should be made of all samples to be compared—one that will bring out the aromatic and subdue the alcoholic constituents. When this has been properly done, an estimation can be made much more readily than by tasting the crude spice in powder. It requires practice to make the method available for the purposes of comparison, but one soon becomes quite expert. It is well known that pungent substances, such as pepper, obscure any gustatory sensation, therefore a solution of pepper must be highly diluted to make it serviceable. It should be said that tastes do not develop promptly. In some cases a considerable time must be allowed to elapse before the taste is well developed. This is doubtless due to the slow miscibility of the sapid and pungent substances. At best the process is to be considered only as a preliminary one for quickly eliminating substandard materials. It is this preliminary test that would be so useful were it made practical for rapidly selecting from a given number of samples those inferior ones that should be set aside for more careful examination by more elaborate methods. As has been stated, the microscope quickly identifies a spice adulterated with foreign material, but a substandard spice, containing all the microscopical elements of the standard material, the microscope is powerless to detect.